UTILIZATION OF WASTE

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GLASS COATINGS BASED ON ELECTRIC PRECIPITATOR DUST

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Compositions based on electric precipitator dust are developed for producing thin films on sheet glass with heat-shielding and decorative properties. A method for conversion of the pigment in the form of solid waste into a solution is developed. The obtained films absorb 39 – 25% thermal radiation in the IR spectrum range.

Production of transparent coatings with decorative and heat-shielding characteristics using the sol-gel technology makes it possible to expand the product range and to lower the production cost of coated glass due to application of inexpensive materials and equipment and an insignificant energy consumption. The advantage of this technology consists in the possibility of developing conditions for interaction of the components at the level of molecular dispersion under low temperatures (300 – 400°C) [1, 2].

In modifying a silicate glass surface with film-forming solutions (FFS), it is possible to use galvanic, metallurgical, and other industrial waste as pigments [3]. The main requirement imposed on such materials is the absence or insignificant content of insoluble residue, since the latter can lead to difficulties in converting the material into a solution.

The purpose of the present study was to investigate the possibility of obtaining heat-shielding and decorative coatings on sheet glass based on the sol-gel technology using electric precipitator dust, which is iron-containing waste generated at the Oskol'skii Electrometallurgy Works.

It was earlier established that the main crystalline phase of this waste is represented by magnetite Fe_3O_4 [4]. Since the production of heat-absorbing coatings on sheet glass was based on the sol-gel technology, we first had to develop a method for converting a solid pigment into a solution, to select a solvent, and to determine the optimum ratio between them. The solvents investigated were both organic and inorganic acids: hydrochloric, nitric, sulfuric, acetic, oxalic, and oleic.

The experiments demonstrated that the most effective solvent was concentrated hydrochloric acid. Dust dissolves in it under normal conditions without a residue. An interesting result was observed in dissolution in acetic and oxalic acids. The dissolution was not complete, but part of the solution transformed into a gel-like material. Accordingly, three acids were selected for further studies: concentrated hydrochloric, glacial acetic, and a concentrated solution of oxalic acid in distilled water.

To select the optimum ratio between the solvent and the precipitator dust, as well as the dissolution duration, a series of experiential solutions was prepared. At the same time the salt density in the course of dissolution was measured. The highest density was registered in iron chloride solutions, in which a density jump (from 1230 to 1263 kg/m^3) was registered on the sixth day of dissolution. An extension of the dissolution duration beyond 6 days had virtually no effect on the dust dissolution process. The dissolution of the modifier in organic acids was less intense. Thus, oxalates exhibited the least intense light yellow tinting of the solution. Acetates depending on the solvent quantity changed their color from a light claret to a saturated claret color. The most intense tinting was manifested in chlorides: from a clear solution (3 days) to an opaque brick color (7 days).

The optimum dust-to-solvent ratio was 1 : 4 (Table 1), as the most complete dissolution of dust occurred under this ratio, which later made it possible to obtain films with better heat-shielding properties. The salt was filtered and used during the rest of the experiments.

The following components were used for FFS preparation in this work: tetraethoxysilane $Si(OC_2H_5)_4$ to introduce SiO_2, which is the glass-forming agent for the film matrix; solutions of precipitator dust in various acids as modifiers of film properties, and ethyl alcohol C_2H_5OH as a solvent.

The calculation of the oxide content and the ratio between the oxides in an FFS was performed on a computer. The molar ratio between iron oxide and silicon oxide varied within the limits from 25 : 75 to 40 : 60%. The total weight content of iron and silicon oxides was equal to 2.5 and 5.0%. Table 2 lists the compositions of the FFS based on iron chlorides.

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TABLE 1

Solution*	Quantity of acid, liters	Dust : solvent ratio	Density, kg/m^3
1	0.100	1 : 4	1350
2	0.150	1 : 6	1300
3	0.150	1 : 6	1297
4	0.175	1 : 7	1277
5	0.175	1 : 7	1280

* Dust quantity in all cases was 0.025 kg.

TABLE 2

Fe : Si molar ratio, %	Total weight content of iron and silicon oxides, %	Quantity, ml		
		$Si(OC_2H_5)_4$	C_2H_5OH	$FeCl_3$
25 : 75	2.5	1.23	30.54	2.31
	5.0	2.46	29.74	4.59
30 : 70	2.5	1.08	30.54	2.58
	5.0	2.16	29.74	5.19
35 : 65	2.5	0.95	30.80	1.25
	5.0	1.91	30.10	1.91
40 : 60	2.5	0.83	30.80	1.35
	5.0	1.67	30.10	2.70

A shift in the molar ratio toward a content of iron oxide of more than 40% did not allow for getting a high-quality film due to the fast gel formation in the FFS. Introduction of less than 25% salt yielded weakly tinted solutions, which in the end deteriorated the heat-shielding and decorative properties of the film.

Increase in the weight content of oxides in an FFS above 10% also facilitated fast formation of gel in the solution and a loss of its service properties. Based on the selected ratios, stable work solutions were obtained, which retained their properties for 8 months or longer.

The coatings were deposited on a substrate of sheet glass by the immersion method at a rate of 1.6 mm/sec on a laboratory plant. This method yielded thin and uniform films with good adhesion.

The heat treatment conditions were selected in a laboratory muffle furnace within a temperature interval of 150 – 450°C. With heat treatment below 300°C, the coating remained soft, and adhesion to the substrate was missing. At a temperature of 400°C the coating burned out. The best coatings were obtained under a temperature of 350°C with an exposure duration of 10 min. If the exposure duration was shortened, the coating did not have time to acquire the necessary strength of adhesion, and a longer exposure resulted in partial burning of the film. The samples with deposited coatings were placed in a heated furnace. Films based on acetates and oxalates had weak tinting and therefore were not used in the further experiments.

The optical properties of coated glasses were studied with a Specord 75 IR instrument in the IR (2500 – 2800 nm) and visible range (400 – 750 nm) using a SF-26 spectrophotometer. The microhardness of the samples with film coating was tested by the Vickers method on a PMT-3 microhardness meter under a 100 g load and a 5 sec exposure. The total light transmission was determined on a POS-1 instrument. The reflection coefficient was measured using a FB-2 photoelectric luster meter taking the mirror reflection as 100%. The physical and optical properties of the experimental samples are listed in Table 3.

TABLE 3

Fe : Si molar ratio, %	Total weight content of iron and silicon oxides, %	Glass properties			
		micro-hardness, MPa	total light transmission, %	reflection coefficient, %	light transmission in IR range
25 : 75	2.5	4836.1	85.5	17	75
	5.0	5573.8	84.1	19	72
30 : 70	2.5	5202.9	85.5	18	72
	5.0	5573.8	81.0	19	70
35 : 65	2.5	4836.2	86.2	20	68
	5.0	5202.9	84.7	22	66
40 : 60	2.5	3049.2	83.3	21	65
	5.0	3574.7	82.6	22	61
Sheet glass without coating	–	5933.9	89.0	17	85

The light transmission of the considered samples in the IR range was 61 – 75% depending on the composition and the iron oxide content in the film. Glasses transmitting up to 60% light in the IR range are regarded as heat-shielding. Variation of the Fe : Si ratio in the film composition had an effect on the microhardness of the coating. Thus, as the molar content of iron oxide grew, the coating became softer. However, an increase in the mass content of oxides from 2.5 to 5.0% to a certain extent compensated for this shortage. Introduction of Fe_2O_3 into the film composition increased the reflection coefficient of the coating by 3 – 5% on the average, compared with the standard sheet glass.

As a result of the studies performed, thin films based on precipitator dust and having heat-shielding and reflecting properties were obtained. A method for the conversion of solid waste into a solution was developed. It was experimentally confirmed that an increase in the weight content of iron oxide in an FFS leads to fast gel formation in the solution (2 – 3 days), and an increase in the iron oxide content in the FFS up to 40% makes it possible to extend the service life of the solution up to 8 months with a simultaneous improvement of the heat-shielding properties of the film. The resulting coatings have sufficiently good heat-shielding characteristics and the proposed technology is simple to use and is not expensive.

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